1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 2

BIOMATERIAL (FISH) SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

G. C. RONAN, DIRECTOR
Laboratory Services Branch
Ministry of the Environment

1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

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Inorganic Trace Contaminants Section
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INORGANIC TRACE CONTAMINANTS SECTION

SUMMARY

I. Introduction

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robins. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

II. Quality Control and Assurance

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

A. Quality Control

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

B. Quality Assurance

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

$$sd = sqrt[\{(sumx2 - (sumx)2\}/n/(n-1)] \quad .....I$$

$$sd = sqrt(sumd2/2n) \quad .....II$$

where : x = the individual values; n = the number of events
d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

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2. Biomaterials

2.1 Fish and Biota

Fish samples are collected and frozen until they can be prepared for analysis. In most cases a filet is taken, ground thoroughly and refrozen as necessary. Other smaller biotic samples are handled as is appropriate to the sample. QA samples consist of composited fish tissue or other referenced material.

TABLE 2.1

Parameter	Collection Device	Preparation	Analysis
Metals	Plastic bags	Acid digest	AAS, ICP-AES
Mercury	Plastic bags	Acid digest	Cold Vapour AAS
Hydride Metals	Plastic bags	Acid digest	AAS

2.2 Fish and Biota Quality Assurance

Sample duplicates are prepared by taking a second aliquot from the prepared sample.

Reagent blanks are analysed with each analytical run. There are sufficient variations in the digestion acid lots that only one lot should be used in any one analytical run.

Matrix matched between-run composite samples are prepared by collecting samples in a large container. New composites are collected as the first is depleted or as the stability period expires. These composites may be spiked as necessary to provide a measureable level of analyte.

Table 2.2 gives the sample designations for the QA materials used for Fish and Biota analyses, the nature of the sample and the parameters for which it is used as a control.

TABLE 2.2

Sample Designation	Type	Parameter
con 683	Composite in-house fish	Metals, Hg
con 386	Composite in-house fish	Metals, Hg
fc-2	In-house whole fish homogenized	Metals, Hg
fc-1	In-house whole fish homogenized, spiked	Metals, Hg
TORT-1	NRC reference lobster	Metals, Hg
EPA 1650	EPA freeze dried fish	Metals, Hg

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Aluminum TEST CODE: ALUT SAMPLE TYPE: Fish
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE: 550AA1
REVISION NO: 84-1 DATE: January, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 g (wet)
Container- Whirl Pac (for heavy metals only)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted- 100
Procedure- Weigh accurately approx. 2.5 g of a snipped sample (See Remarks) into a calibrated 25 x 200 mm Pyrex test tube and add 7 ml HNO_3 and 1.5 ml $HClO_4$.
Prepare a batch of 40 tubes in this manner, including duplicates and spikes, and load into an aluminum heating block containing 40 holes of appropriate size.
Heat tubes at 120°C overnight (about 15 h). Increase temperature gradually over a 1 hour period to 180°C.
Evaporate down to approx. 0.5 ml. Add double distilled water and and dilute to 25 ml. Mix throughly and submit to AAS for analysis.

INTERFERENCES: None

REPORTING RESULTS:One place after decimal if <10 ug/g;whole no if >10
INSTRUMENTATION: Perkin Elmer 5000 AAS with autosampler and PET computer interface for data message, storage and transfer to lab central data handling computer.
Calibration Range: 0 to 20 ug/ml
Resolution: 0.01
Sensitivity: 1.0 ug/ml
Instrument Detection Limit: 0.04 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1.0000 to 200.0 mg/kg
Accuracy-
Precision of Controls-

	A	B
mean	2.747	9.102
std. dev.	0.6167	0.4111
R.S.D.	22.45	4.52

Precision of Duplicates-low range mid range high range
s.d.
mean
W T

CONTROL LIMITS:

REMARKS: Snip samples are portions of the received fillet cut with scissors.

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN FISH

Range = 1.0000to 200.0 mg/kg

IN - RUN DUPLICATES

Range	<1.0000	1.0000to40.00	40.00 to100.00	100.00to200.0	>200.0
no.	0	0	0	0	0
s.w.		0.0000	0.0000	0.0000	
mean		0.0000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
fc-1	0	0.000	0.0000	0.00
fc-2	0	0.000	0.0000	0.00
TORT-1	0	0.000	0.0000	0.00
epa-1650	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/03/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cadmium TEST CODE: CDUT SAMPLE TYPE: Fish
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE: 550AA1
REVISION NO: 84-1 DATE: January, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 g (wet)
Container- Whirl Pac (for heavy metals only)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted- 100
Procedure- Weigh accurately approx. 2.5 g of a snipped sample (See Remarks) into a calibrated 25 x 200 mm Pyrex test tube and add 7 ml HNO3 and 1.5 ml HClO4.
Prepare a batch of 40 tubes in this manner, including duplicates and spikes, and load into an aluminum heating block containing 40 holes of appropriate size.
Heat tubes at 120°C overnight (about 15 h). Increase temperature gradually over a 1 hour period to 180°C.
Evaporate down to approx. 0.5 ml. Add double distilled water and and dilute to 25 ml. Mix throughly and submit to AAS for analysis.

INTERFERENCES: None

REPORTING RESULTS:One place after decimal if <10 ug/g;whole no if >10
INSTRUMENTATION: Perkin Elmer 5000 AAS with autosampler and PET computer interface for data message, storage and transfer to lab central data handling computer.
Calibration Range: 0.0 to 0.5 ug/ml
Resolution: 0.01
Sensitivity: 0.025 ug/ml
Instrument Detection Limit: 0.004 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.04 to 5.0 mg/kg
Accuracy-
Precision of Controls-

	A	B
mean	.055mg/kg	.195mg/kg
std. dev.	.022mg/kg	.043mg/kg
R.S.D.	39 %	22 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	ND	ND	ND
mean	ND	ND	ND

W .05 mg/kg T .20 mg/kg

CONTROL LIMITS:

REMARKS: Snip samples are portions of the received fillet cut with scissors.
Graphite furnace can be used when lower detection limits are required.

SUMMARY REPORT OF QUALITY CONTROL DATA

CD-FLAME IN FISH

Range = .04 to 5.0 mg/kg

IN - RUN DUPLICATES

Range	<.04	.04 to1.00	1.00 to2.50	2.50 to5.00	>5.00
no.	16	0	0	0	0
s.w.		0	0	0	
mean		0	0	0	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
fc-1	16	0.055	0.0217	39.45
fc-2	11	0.084	0.0156	18.57
TORT-1	7	24.976	1.4792	5.92
epa-1650	6	0.195	0.0426	21.85

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Chromium TEST CODE: CRUT SAMPLE TYPE: Fish
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE: 550AA1
REVISION NO: 84-1 DATE: January, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 g (wet)
Container- Whirl Pac (for heavy metals only)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted- 100
Procedure- Weigh accurately approx. 2.5 g of a snipped sample (See Remarks) into a calibrated 25 x 200 mm Pyrex test tube and add 7 ml HNO_3 and 1.5 ml $HClO_4$.
Prepare a batch of 40 tubes in this manner, including duplicates and spikes, and load into an aluminum heating block containing 40 holes of appropriate size.
Heat tubes at 120°C overnight (about 15 h). Increase temperature gradually over a 1 hour period to 180°C.
Evaporate down to approx. 0.5 ml. Add double distilled water and and dilute to 25 ml. Mix throughly and submit to AAS for analysis.

INTERFERENCES: None

REPORTING RESULTS:One place after decimal if <10 ug/g;whole no if >10
INSTRUMENTATION: Perkin Elmer 5000 AAS with autosampler and PET computer interface for data message, storage and transfer to lab central data handling computer.
Calibration Range: 0 to 5 ug/ml
Resolution:.01 ug/ml
Sensitivity:
Instrument Detection Limit:.01 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1000 to 50.0 mg/kg
Accuracy-
Precision of Controls-

	A	B
mean	2.639	6.107
std. dev.	0.3925	2.6027
R.S.D.	14.87	42.62

Precision of Duplicates-low range mid range high range
s.d.
mean
W T

CONTROL LIMITS:

REMARKS: Snip samples are portions of the received fillet cut with scissors.

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN FISH

Range = 0.1000to 50.0 mg/kg

IN - RUN DUPLICATES

Range	<0.1000	0.1000to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	0	0	0	0	0
s.w.		0.0000	0.0000	0.0000	
mean		0.0000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
fc-1	16	2.639	0.3925	14.87
fc-2	0	0.000	0.0000	0.00
TORT-1	4	3.450	1.1121	32.23
epa-1650	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Copper TEST CODE: CUUT SAMPLE TYPE: Fish
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE: 550AA1
REVISION NO: 84-1 DATE: January, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 g (wet)
Container- Whirl Pac (for heavy metals only)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted- 100
Procedure- Weigh accurately approx. 2.5 g of a snipped sample (See Remarks) into a calibrated 25 x 200 mm Pyrex test tube and add 7 ml HNO_3 and 1.5 ml $HClO_4$.
Prepare a batch of 40 tubes in this manner, including duplicates and spikes, and load into an aluminum heating block containing 40 holes of appropriate size.
Heat tubes at 120°C overnight (about 15 h). Increase temperature gradually over a 1 hour period to 180°C.
Evaporate down to approx. 0.5 ml. Add double distilled water and and dilute to 25 ml. Mix throughly and submit to AAS for analysis.

INTERFERENCES: None

REPORTING RESULTS:One place after decimal if <10 ug/g;whole no if >10
INSTRUMENTATION: Perkin Elmer 5000 AAS with autosampler and PET computer interface for data message, storage and transfer to lab central data handling computer.
Calibration Range: 0.0 to 5.0 ug/ml
Resolution: 0.01
Sensitivity: 0.09 ug/ml
Instrument Detection Limit: 0.02 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1000 to 50.0 mg/kg
Accuracy-
Precision of Controls-

	A	B
mean	1.615	5.521
std. dev.	0.2392	0.3376
R.S.D.	14.81	6.11

Precision of Duplicates-low range mid range high range
s.d. 0.1118
mean 0.8060
W .01 mg/kg T .05 mg/kg

CONTROL LIMITS:

REMARKS: Snip samples are portions of the received fillet cut with scissors.

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER IN FISH

Range = 0.1000to 50.0 mg/kg

IN - RUN DUPLICATES

Range	<0.1000	0.1000to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	0	15	0	0	0
s.w.		0.1118	0.0000	0.0000	
mean		0.8060	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
fc-1	30	1.615	0.2392	14.81
fc-2	0	0.000	0.0000	0.00
TORT-1	6	375.833	16.4489	4.38
epa-1650	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/03/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Lead TEST CODE: PBUT SAMPLE TYPE: Fish
UNIT: Biomaterials SUPERVISOR: R. Sadana)

METHOD CODE: 550AA1
REVISION NO: 84-1 DATE: January, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 g (wet)
Container- Whirl Pac (for heavy metals only)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted- 100
Procedure- Weigh accurately approx. 2.5 g of a snipped sample (See Remarks) into a calibrated 25 x 200 mm Pyrex test tube and add 7 ml HNO_3 and 1.5 ml $HClO_4$.
Prepare a batch of 40 tubes in this manner, including duplicates and spikes, and load into an aluminum heating block containing 40 holes of appropriate size.
Heat tubes at 120°C overnight (about 15 h). Increase temperature gradually over a 1 hour period to 180°C.
Evaporate down to approx. 0.5 ml. Add double distilled water and 0.5 ml of 5% potassium solution to each tube and dilute to 25 ml.
Dilute sample 5X and submit to graphite furnace for analysis.
INTERFERENCES: None

REPORTING RESULTS:One place after decimal if <10 ug/g;whole no if >10
INSTRUMENTATION: Perkin Elmer 2380 or 603 AA Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler.

Calibration Range: 0 to .100 ug/ml
Resolution: 0.001
Sensitivity: 0.030 ug/ml = .200 Abs.
Instrument Detection Limit: 0.001 ug/ml

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.05 to 1.00 mg/kg
Accuracy-
Precision of Controls-

	A	B
mean	0.408	0.104
std. dev.	0.1810	0.0641
R.S.D.	44.36	61.63

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0277	0.0939	
mean	0.0730	0.3180	

W T

CONTROL LIMITS:

REMARKS: Snip samples are portions of the received fillet cut with scissors.

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD IN FISH

Range = 0.0100to 1.0 mg/kg

IN - RUN DUPLICATES

Range	<0.0100	0.0100to0.20	0.20 to0.50	0.50 to1.0	>1.0
no.	0	18	2	0	0
s.w.		0.0277	0.0939	0.0000	
mean		0.0730	0.3180	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
fc-1	27	0.408	0.1810	44.36
fc-2	0	0.000	0.0000	0.00
TORT-1	0	0.000	0.0000	0.00
epa-1650	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Manganese TEST CODE: MNUT SAMPLE TYPE: Fish
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE: 550AA1
REVISION NO: 84-1 DATE: January, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 g (wet)
Container- Whirl Pac (for heavy metals only)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted- 100
Procedure- Weigh accurately approx. 2.5 g of a snipped sample (See Remarks) into a calibrated 25 x 200 mm Pyrex test tube and add 7 ml HNO_3 and 1.5 ml $HClO_4$.
Prepare a batch of 40 tubes in this manner, including duplicates and spikes, and load into an aluminum heating block containing 40 holes of appropriate size.
Heat tubes at 120°C overnight (about 15 h). Increase temperature gradually over a 1 hour period to 180°C.
Evaporate down to approx. 0.5 ml. Add double distilled water and and dilute to 25 ml. Mix throughly and submit to AAS for analysis.

INTERFERENCES: None

REPORTING RESULTS:One place after decimal if <10 ug/g;whole no if >10
INSTRUMENTATION: Perkin Elmer 5000 AAS with autosampler and PET computer interface for data message, storage and transfer to lab central data handling computer.
Calibration Range: 0.0 to 5 ug/ml
Resolution: 0.01
Sensitivity: 0.055 ug/ml
Instrument Detection Limit: 0.04 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1000 to 50 mg/kg
Accuracy-
Precision of Controls-

	A	B
mean	2.739	1.032
std. dev.	0.2937	0.3195
R.S.D.	10.72	30.96

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.1556	ND	ND
mean	0.5000	ND	ND

W T

CONTROL LIMITS:

REMARKS: Snip samples are portions of the received fillet cut with scissors.

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN FISH

Range = 0.1000to 50.0 mg/kg

IN - RUN DUPLICATES

Range	<0.1000	0.1000to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	0	1	0	0	0
s.w.		0.1556	0.0000	0.0000	
mean		0.5000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
fc-1	14	2.739	0.2937	10.72
fc-2	0	0.000	0.0000	0.00
TORT-1	6	21.133	0.6593	3.12
epa-1650	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Mercury TEST CODE:HGUT SAMPLE TYPE: Fish
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE: TYPE: Flameless AAS
REVISION NO: Original DATE: May, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approx. 50 g
Container- Glass vials
Preservative- Freezing
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Weigh approx. 0.250 g of ground fish tissue into a 50 ml Folin-Wu digestion tube. Add 5 ml of acid mixture (4:1 - H_2SO_4:HNO_3) and place the tube in an aluminum hot block placed on a hot plate (approx 220°C). Digest for 16 hrs (overnight). Cool, dilute to 25 ml with distilled water.
Run in batches of 50 or more.

Treat blanks and calibration standards in exactly the same manner. Determine mercury in the entire volume. The measurement step is automated and is based on the evolution of atomic vapour of mercury (wavelength - 254nm) by the addition of a reducing agent.
INTERFERENCES: Water vapour; organic solvents.

REPORTING RESULTS:Two significant figures (ug/g).
INSTRUMENTATION: Automated sampler and peristaltic pump (Technicon or Gilson). Laboratory Data Control U.V. monitor (Pharmacia or Milton-Roy).
Calibration Range: 0 - 16 ng/ml
Resolution: 0.4 ng/ml (one division on recorder chart paper)
Sensitivity:1.0 ng/ml reads 0.05 Absorbance (2.5 divs on chart)
Instrument Detection Limit: 0.1 ng/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0 - 2.0 ug/g
Accuracy- 110% at 0.94 ug/g (NBS Albacore tuna)
Precision of Controls-

	A	B
mean	.216mg/kg	.785mg/kg
std. dev.	.015mg/kg	.037mg/kg
R.S.D.	7.0 %	4.7 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	.027	.050	.034
mean	.177	.601	1.23

W 0.01 µg/g T 0.05 µg/g

CONTROL LIMITS:

REMARKS:

- Detection Limit - 2x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MERCURY IN FISH

Range = 0.0100to 2.0 ug/g

IN - RUN DUPLICATES

Range	<0.0100	0.0100to0.40	0.40 to1.00	1.00 to2.0	>2.0
no.	1	92	21	9	2
s.w.		0.0272	0.0496	0.0337	
mean		0.1770	0.6010	1.2340	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
con 683	127	0.216	0.0151	6.99
con 386	90	0.785	0.0368	4.69

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0	0

DATE 86/12/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nickel TEST CODE: NIUT SAMPLE TYPE: Fish
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE: 550AA1
REVISION NO: 1 DATE: January, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 g (wet)
Container- Whirl Pac (for heavy metals only)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted- 100
Procedure- Weigh accurately approx. 2.5 g of a snipped sample (See Remarks) into a calibrated 25 x 200 mm Pyrex test tube and add 7 ml HNO_3 and 1.5 ml $HClO_4$.
Prepare a batch of 40 tubes in this manner, including duplicates and spikes, and load into an aluminum heating block containing 40 holes of appropriate size.
Heat tubes at 120°C overnight (about 15 h). Increase temperature gradually over a 1 hour period to 180°C.
Evaporate down to approx. 0.5 ml. Add double distilled water and and dilute to 25 ml. Mix throughly and submit to AAS for analysis.

INTERFERENCES: None

REPORTING RESULTS:One place after decimal if <10 ug/g;whole no if >10
INSTRUMENTATION: Perkin Elmer 5000 AAS with autosampler and PET computer interface for data message, storage and transfer to lab central data handling computer.
Calibration Range: 0.0 to 5.0 ug/ml
Resolution: 0.01
Sensitivity: 0.15 ug/ml
Instrument Detection Limit: 0.04 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1000 to 50.0 mg/kg
Accuracy-
Precision of Controls-

	A	B
mean	1.257	0.851
std. dev.	0.4381	0.1558
R.S.D.	34.85	18.31

Precision of Duplicates-low range mid range high range
s.d. 0.1939
mean 0.8040
W .02 mg/kg T .10 mg/kg

CONTROL LIMITS:

REMARKS: Snip samples are portions of the received fillet cut with scissors.

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN FISH

Range = 0.1000to 50.0 mg/kg

IN - RUN DUPLICATES

Range	<0.1000	0.1000to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	0	4	0	0	0
s.w.		0.1939	0.0000	0.0000	
mean		0.8040	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
fc-1	26	1.257	0.4381	34.85
fc-2	0	0.000	0.0000	0.00
TORT-1	6	3.750	0.9022	24.06
epa-1650	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/03/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zinc TEST CODE: ZNUT SAMPLE TYPE: Fish
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE: 550AA1
REVISION NO: 84-1 DATE: January, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 g (wet)
Container- Whirl Pac (for heavy metals only)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted- 100
Procedure- Weigh accurately approx. 2.5 g of a snipped sample (See Remarks) into a calibrated 25 x 200 mm Pyrex test tube and add 7 ml HNO_3 and 1.5 ml $HClO_4$.
Prepare a batch of 40 tubes in this manner, including duplicates and spikes, and load into an aluminum heating block containing 40 holes of appropriate size.
Heat tubes at 120°C overnight (about 15 h). Increase temperature gradually over a 1 hour period to 180°C.
Evaporate down to approx. 0.5 ml. Add double distilled water and and dilute to 25 ml. Mix throughly and submit to AAS for analysis.

INTERFERENCES: None

REPORTING RESULTS:One place after decimal if <10 ug/g;whole no if >10
INSTRUMENTATION: Perkin Elmer 5000 AAS with autosampler and PET computer interface for data message, storage and transfer to lab central data handling computer.
Calibration Range: 0.0 to 5.0 ug/ml
Resolution: 0.01
Sensitivity: 0.18 ug/ml
Instrument Detection Limit: 0.02 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1000 to 50.0 mg/kg
Accuracy-
Precision of Controls-

	A	B
mean	34.880	12.883
std. dev.	3.1565	5.5724
R.S.D.	9.05	43.25

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.3874	1.4922	4.0415
mean	3.9470	17.6000	30.3330

W .1 mg/kg T .5 mg/kg

CONTROL LIMITS:

REMARKS: Snip samples are portions of the received fillet cut with scissors.

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC IN FISH

Range = 0.1000to 50.0 mg/kg

IN - RUN DUPLICATES

Range	<0.1000	0.1000to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	0	10	3	3	0
s.w.		0.3874	1.4922	4.0415	
mean		3.9470	17.6000	30.3330	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
fc-1	30	34.880	3.1565	9.05
fc-2	0	0.000	0.0000	0.00
TORT-1	6	156.167	3.6009	2.31
epa-1650	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.